NOT MEASUREMENT SENSITIVE

MIL-STD-3010 30 December 2002 SUPERSEDING FED-STD-101C 13 March 1980

DEPARTMENT OF DEFENSE

TEST METHOD STANDARD

TEST PROCEDURES FOR PACKAGING MATERIALS



AMSC N/A AREA PACK

FOREWORD

- 1. This standard is approved for use by all Departments and Agencies of the Department of Defense (DoD).
- 2. This standard was developed to document uniform test methods for the evaluation of materials used in military packaging applications. Reference to the test methods included herein ensures standardization of testing procedures and also eliminates unnecessary repetition of detailed test instructions within each individual packaging material specification.
- 3. The test methods included herein were previously documented in FED-STD-101. FED-STD-101A contained 250 test methods. This number was reduced to 172 in FED-STD-101B, then to 31 in FED-STD-101C, by deleting test methods no longer deemed relevant or as a result of supersession by industry standards. The remaining 31 standard test methods have recently been reviewed to verify current need and extent of usage. This review indicated that only 15 unique test method standards are required to support the testing of materials used in military packaging applications. These 15 test methods have thus been carried over to this Military Standard. A complete chronology of the above-mentioned history of packaging material standard test methods is included herein as Appendix A.
- 4. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Air Warfare Center Aircraft Division, Highway 547, Code 414100B120-3, Lakehurst, NJ 08733-5100, by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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1. SCOPE

1.1 <u>Scope</u>. This document provides a centralized listing of detailed and uniform test methods that have been developed to evaluate relevant properties of materials used in military packaging applications. These standardized test methods may be referenced by number in specific packaging material specifications, as applicable, thus eliminating the need to repetitively detail the standard test method in each material specification.

2. APPLICABLE DOCUMENTS

2.1 <u>General</u>. The documents listed in this section are specified in sections 3, 4, and 5 of this standard. This section does not include documents cited in other sections of this standard or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in section 3, 4, and 5 of this standard, whether or not they are listed.

2.2 Government documents.

2.2.1 <u>Specifications and standards</u>. The following specifications and standards form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS) and supplement thereto, cited in the solicitation (see 6.2).

SPECIFICATIONS

FEDERAL

QQ-S-698 - Steel, Sheet and Strip, Low Carbon.

DEPARTMENT OF DEFENSE

MIL-PRF-131 - Barrier Materials, Watervaporproof, Greaseproof,

Flexible, Heat-Sealable.

MIL-PRF-680 - Solvent, Degreasing.

(Unless otherwise indicated, copies of the above specifications are available from the Standardization Documents Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.3 <u>Non-Government publications</u>. The following documents form a part of this document to the extent specified. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM-D471 - Rubber Property – Effect of Liquids. (DoD adopted)
ASTM-D996 - Standard Terminology of Packaging and Distribution
Environments. (DoD adopted)

(Application for copies should be addressed to the American Society for Testing and Materials (ASTM), 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.4 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. DEFINITIONS

- 3.1 <u>General</u>. Definitions of terms unique to this standard are listed below. Definitions of other terms commonly used in the packaging community may be found in ASTM-D996.
- 3.2 <u>Blocking</u>. Cohesion or adhesion between contiguous layers of similar or dissimilar materials in roll or sheet form which interferes with the satisfactory and efficient use of the material.
 - 3.3 Curl. Tendency of an unrestrained flat strip of material to roll-up on itself.
- 3.4 <u>Leak</u>. Any opening in a container, contrary to design intent, that either allows the contents to escape or permits substances to enter.
- 3.5 <u>Stain</u>. Color changes formed only on a surface without any evidence of rust, pitting, etching, or deterioration of the surface.
- 3.6 <u>Water vapor transmission rate (WVTR)</u>. The mass of water vapor transmitted through a given area of test material in a given time when the test material is maintained at a constant temperature, and when one surface is exposed to very low relative humidity and the other surface to a high relative humidity.

4. GENERAL REQUIREMENTS

- 4.1 <u>Test method format</u>. The standard format used to define packaging material test methods herein is as follows:
 - a. Scope
 - b. Apparatus
 - c. Test specimens
 - d. Test procedure
 - e. Notes
- 4.2 <u>Test report format</u>. Following the conduct of each test, a report shall be prepared. This report shall contain, as a minimum, the following information:
 - a. A statement that the test was conducted in compliance with the procedure(s) detailed herein, or a description of any deviations from same.
 - b. Identification of each specimen/material tested.
 - c. Results of the test.
 - d. An indication of compliance or non-compliance with specification requirements.
- 4.3 <u>Test methods</u>. Each standard test method is described in detail in section 5. The test method numbers have been retained from their originally assigned designations.
- 4.4 <u>Test room conditions</u>. Unless otherwise specified, all testing described herein shall be conducted on test specimens that have been conditioned for at least 24 hours in a test room maintained at $73^{\circ} \pm 3.5^{\circ}$ F and 50 ± 5 percent relative humidity (RH).

5. DETAILED REQUIREMENTS

- 5.1 <u>Dimensional properties test methods</u>.
- 5.1.1 Test Method 1003 Thickness.
- 5.1.1.1 <u>Scope</u>. This test procedure details a method for determining the thickness of specimens using a dial micrometer on a test stand. It is intended for use on flexible packaging materials and on cushioning materials.

5.1.1.2 Apparatus.

- 5.1.1.2.1 A dial micrometer reading in thousandths of an inch and calibrated to be accurate within 0.5 percent or one dial division, whichever is greater. The micrometer shall be securely mounted on a rigid test stand.
- 5.1.1.2.2 A flat metal plate measuring 6 inches by 6 inches and weighing two and one-half pounds, thus creating a load of 0.10 pounds per square inch on a test sample measuring 5 inches by 5 inches.
- 5.1.1.3 <u>Test specimens</u>. Unless otherwise specified, five specimens, each measuring 5 inches by 5 inches, shall be selected at random from representative locations of the material being evaluated.
- 5.1.1.4 <u>Test procedure</u>. The dial of the micrometer shall be zeroed against the weight plate while the plate rests on the base of the test stand. The plate shall then be carefully removed from the stand without disturbing the zero setting of the micrometer. The test specimen shall be placed on the base of the stand so as to be centered beneath the micrometer dial. The weighted plate shall then be gently placed on the test specimen so that it is centered over the specimen. After thirty seconds, the micrometer shall be read to the nearest 0.001 inch, thus measuring the thickness of the test specimen.

- 5.2 Strength and elastic properties test methods.
- 5.2.1 <u>Test Method 2015 Curl.</u>
- 5.2.1.1 <u>Scope</u>. This test procedure details a method to determine the curling tendencies of barrier materials when exposed to normal or elevated temperatures.
 - 5.2.1.2 Apparatus.
 - 5.2.1.2.1 A drying oven capable of maintaining a temperature of $160^{\circ} \pm 2^{\circ}$ F.
- 5.2.1.3 <u>Test specimens</u>. Specimens shall be selected at random to adequately represent any variation of the material being evaluated. Each specimen shall measure $12 \pm 1/16$ inch by 36 $\pm 1/16$ inch. Six specimens shall be tested, three taken from each principal direction of the material.
- 5.2.1.4 Test procedure. The specimens shall be placed on a horizontal surface. A weight shall be placed over one 12-inch edge of the specimen to restrain its motion. After thirty minutes, the horizontal distance (length) between the restrained edge of the specimen and the free edge shall be measured to the nearest one-sixteenth of an inch to determine the curling tendencies of the material. The specimens shall then be placed in a forced draft oven maintained at $160^{\circ} \pm 2^{\circ}$ F for 168 hours. The specimens shall be arranged in the oven to permit free circulation of air over the specimens throughout the exposure. After removal from the oven, the specimens shall be allowed to cool in the test room for two hours. Measurements as described above shall be taken on the specimens to redetermine the curling tendencies of the material after exposure to elevated temperatures.
 - 5.2.1.5 Notes. Curling shall be identified as a percentage calculated as follows:

Original length of sample (inches) minus length of sample after test (inches) divided by the original length of sample (inches) $\times 100 = \text{Curling Percentage}$.

- 5.2.2 <u>Test Method 2017 Flexing Procedure for Barrier Materials.</u>
- 5.2.2.1 <u>Scope</u>. This test procedure details standard methods for repeatedly flexing barrier materials to simulate their use and handling.
 - 5.2.2.2 Apparatus.
- 5.2.2.2.1 A Gelbo Flex-Tester or equivalent shall be used to provide a standard flexing motion to materials being tested. The testing apparatus shall consist essentially of a 3-1/2 inch diameter stationary head and a 3-1/2 inch diameter movable head spaced at a distance of 7 inches from face-to-face at the starting position of the flexing stroke. The specimen supporting shoulders on each head are 1/2 inch wide. The motion of the movable head is controlled by a grooved shaft to which it is attached. For the full stroke operation the groove is so designed as to give a twisting motion of 440° in the first 3-1/2 inches of the stroke of the movable head followed by a straight horizontal motion of 2-1/2 inches. The motions of the movable head are uniform except for that portion where the rotary motion is changing to straight translational motion. The motion of the machine is reciprocal, a full cycle consisting of the forward and return strokes. For an alternate short stroke operation, the movable head travels only 3-1/4 inches in each direction in such a manner that a twisting motion of only 400° is imparted to the material. The flexing speed for all materials shall be 40 cycles per minute. In the event an equivalent test is used, proof of equivalent motion to that of the Gelbo Flex-Tester shall be presented to the qualifying activity for their approval of the substitute equipment.
 - 5.2.2.2.2 A heat sealer equipped with controls for temperature, dwell time, and pressure.
- 5.2.2.3 Test specimens. Four 12 by 8 inch specimens shall be cut from the barrier material, two in each principal direction. Four additional 13 by 9 inch specimens shall be cut out, two in each principal direction, and shall be aged by exposing the specimens in an atmosphere of 80 to 85 percent relative humidity at 160° ± 2°F for 72 consecutive hours. This relative humidity can be maintained at 160°F over a saturated solution of ammonium sulfate (85 grams per 100 ml water) in a closed vessel. At the completion of the aging exposure, the test specimens shall be returned to test room conditions for 4 hours and then trimmed to produce four aged specimens each 12 by 8 inches. Each test specimen, unaged and aged, shall be prepared for flexing by applying a one-half inch heat seal (or joining by another appropriate means) to the two shorter edges of the sheet, thus producing an approximate 3-1/2 inch diameter cylinder or sleeve 8 inches long.
- 5.2.2.4 Test procedure. Unless otherwise specified, the flexing procedure shall be performed in a test room maintained at $73^{\circ} \pm 3.5^{\circ}$ F and 50 ± 5 percent relative humidity. The sleeve, in cylindrical form, shall be positioned and clamped on the circular heads of the flexing apparatus. The drive shaft of the Flex machine shall be at dead center (i.e., perfectly horizontal)

before flexing is initiated. Each sample shall be flexed for 20 cycles using the full stroke or short stroke. Unless otherwise specified, the full stroke shall be used. The water vapor transmission rate after flexing shall then be determined by forming the specimen into a pouch and testing according to Method 3030 (see 5.3.6).

- 5.2.3 <u>Test Method 2024 Heat-sealed Seam Strength.</u>
- 5.2.3.1 <u>Scope</u>. This test procedure details a method to assess the adequacy of heat seals made on sheet materials and those made in the fabrication of pouches.
 - 5.2.3.2 Apparatus.
- 5.2.3.2.1 Appropriate heat-sealing equipment with accurate controls of temperature, pressure, dwell time, or other sealing criteria required to fuse heat seals of reproducible quality.
- 5.2.3.2.2 One-inch wide clamps from which test weights can be suspended. Unless otherwise specified, the test weight shall be 3-1/2 pounds.
 - 5.2.3.2.3 A test frame to allow the weighted load to act freely on the test specimen.
 - 5.2.3.3 <u>Test specimens</u>.
- 5.2.3.3.1 Sheet materials. A 6 by 12 inch representative section of the material being evaluated shall be folded in half with the crease parallel to the long axis. The open unfolded length shall be heat-sealed together using heat-sealing equipment adjusted appropriately for the material. A line shall be drawn on the back of the specimen while the specimen is in the sealer using a sharp graphite pencil. One-inch wide strips shall be cut off from each end of the heat seal. The fold shall then be cut off from the specimen. After allowing the seals to cool for one hour, three one-inch wide test specimens shall then be cut from the remaining heat seal.
- 5.2.3.3.2 <u>Fabricated bags and pouches</u>. Three one-inch wide heat seal specimens shall be selected from representative locations of the pouch. Areas within one inch of the pouch opening and those areas containing a double seam shall be avoided when obtaining test specimens.
- 5.2.3.4 <u>Test procedure</u>. Unless otherwise specified, the test shall be performed at test room conditions. Fasten one end of the test specimen to the test frame and allow the rest of the specimen to hang free. Carefully and without impact loading, attach the weighted clamp to the lower end of the specimen so the weight is suspended by the specimen. Unless otherwise specified, the weight shall remain freely suspended for 5 minutes. Then remove the weight and measure to 1/32 of an inch the extent to which the heat-sealed seam opened within the marked edges of the heat-sealed area.

- 5.2.4 Test Method 2065 Puncture Resistance.
- 5.2.4.1 <u>Scope</u>. This procedure details a test method for determining the puncture resistance of flexible barrier materials used in military packaging.
 - 5.2.4.2 Apparatus.
 - 5.2.4.2.1 An Instron Tensile Tester equipped with a compression head.
- 5.2.4.2.2 Three compression cells having ranges of 1 to 50 pounds, 20 to 1000 pounds, and 100 to 5000 pounds.
- 5.2.4.2.3 A compression load cell table with accompanying adapter collar compatible with the compression cell being used.
 - 5.2.4.2.4 A test specimen holding fixture as shown on Figure 1.
- 5.2.4.3 <u>Test specimens</u>. Five specimens, each measuring 2 by 2 inches, shall be selected at random from a representative area of the material being evaluated that is free of obvious flaws or defects.

5.2.4.4 <u>Test procedure</u>.

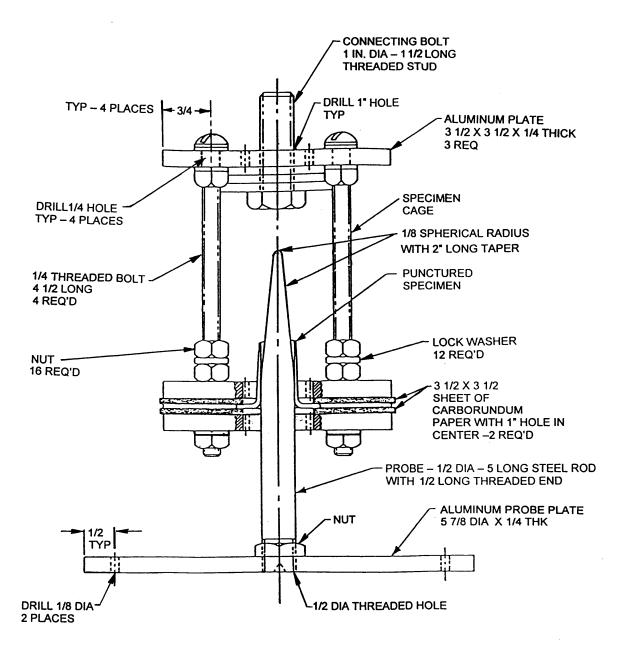
- 5.2.4.4.1 Preparation of measurement apparatus. Select a compression load cell covering the anticipated load required to puncture the test specimen. Lower the cell into the compression block with the power coupling side down. Based upon the load requirements, select the proper screw collar adapter to fit the cell and the compression table. Set the probe plate upon the compression table so that the holes are aligned and secure the plate and table together. Screw the probe into the probe plate and secure with a locking nut. Set the Instron Load Selector switch to position 5. With the load weighing system toggle switch in the "off" position connect the adapter wire first to the compression cell. Pass the wire up from behind the instrument and finally over the upper cross member for connection of the captive coupling to the load weighing system input connector. Energize the load weighing system, place the load selector switch to the No. 1 position for C compression cell operation, or to position 2 for D and E cell operation. Place the proper calibration weight on the probe plate, one, 10 or 25 pounds for Cell C, D, and E, respectively, and proceed with instrument calibration. Deenergize the load weighing system. Return the load selector switch to position 5.
- 5.2.4.4.2 <u>Determination of puncture resistance</u>. Testing of each test specimen shall be conducted as follows:

- a. Screw the connecting bolt on the top side of the test specimen holding fixture (see Figure 1) into the mating threaded hole in the center of the compression anvil located centrally and on the underside of the test crosshead.
- b. Loosen the 4 nuts on the bottom of the birdcage specimen holder and slip the specimen between the faces of the carborundum papers and holddown plates. Center the specimen over the holes in the carborundum papers. Fix the specimen tightly into place by screwing down the nuts. Uniform tightening may be accomplished by progressive tightening of opposite pairs of nuts.
- c. Using the crosshead down button, drive the crosshead to a point approximately 1 inch above the probe. Finally, using the manual positioning knob of the selsyn drive, adjust the crosshead such that the specimen test area and the probe tip are separated by 0.10 to 0.20 inch. Zero the gage length control dial.
- d. Using a ruler, determine to the nearest 0.10 inch, the distance from the bottom flange of specimen clamp to the tip of the probe table. Set the gage length return dial to this distance in inches minus 0.2 inch. Set, to the left and in the automatic return position, the toggle switch located directly below the red safety stop button and to the right of the traverse return button. Select the position of the selsyn drive gear box for the proper gear combination which, with the proper clutch level position, will provide a crosshead movement rate of 20 inches per minute.
- e. Select and position in the chart drive gear box a gear combination to provide a chart speed of 20 inches per minute.
- f. Record on the chart the crosshead and chart speeds in inches per minute.
- g. Place the tester load selector switch in a position which shall cover the anticipated load requirements and which, with the previously set chart speed, shall provide a compression loading slope of approximately 45 degrees.
- h. Activate the load weighing system. Energize the chart drive and simultaneously strike the crosshead down button.

CAUTION

Keep the test under constant surveillance to be certain that the compression load is not being exceeded and that the specimen clamp bed plate does not make direct contact with the compression cable.

- i. When the specimen has been punctured, return the crosshead to its preset gage length point. Deactivate the load weighing and chart drive system. Loosen the nuts and remove the spent specimen. Place a new specimen in the specimen holder. Adjust the chart speed and load range, if required, to provide the desired slope on the records. Repeat the test procedure until five specimens have been tested.
- 5.2.4.4.3 <u>Test results</u>. The maximum force in pounds, to the nearest 0.1 pound, shall be obtained from the recorder records for each specimen tested.



NOTES:

- 1. ALL DIMENSIONS ARE IN INCHES.
- 2. ROUGH SURFACES OF CARBORUNDUM PAPER SHALL FACE EACH OTHER.

FIGURE 1. Test specimen holding fixture.

- 5.3 <u>Resistance properties</u>.
- 5.3.1 <u>Test Method 3003 Blocking Resistance</u>.
- 5.3.1.1 <u>Scope</u>. This test procedure details test methods for determining the resistance of packaging materials adhering to similar or dissimilar packaging materials with which they might come in contact during their storage and service life.
 - 5.3.1.2 Apparatus.
 - 5.3.1.2.1 A circulating air oven that can be maintained at temperatures up to $160^{\circ} \pm 2^{\circ}$ F.
 - 5.3.1.2.2 Two resilient pads, each measuring 2 by 2 by 1/8 inches.
 - 5.3.1.2.3 Two glass or smooth flat plates, each measuring 4 x 4 inches.
 - 5.3.1.2.4 A test room maintained at $73^{\circ} \pm 3.5^{\circ}$ F and 50 ± 2 percent relative humidity.
- 5.3.1.3 <u>Test specimens</u>. Eight specimens, each 3 by 3 inches, shall be selected at random from representative locations of the sheet material being tested.
- 5.3.1.4 <u>Test procedure</u>. The test specimens shall be stacked under room conditions in the following sequence:

Bottom smooth flat plate

Resilient pad

One test specimen, face up

One test specimen, face down

One test specimen, face down

One test specimen, face up

A neutral interleaving material (paper or foil)

One test specimen, face up

One test specimen, face down

One test specimen, face down

One test specimen, face up

Top smooth flat plate

All items shall be centered over the member beneath them. A weight sufficient to produce a 3.0 psi load on the specimens shall be placed on the top plate. The entire assembly shall then be placed in a circulating air oven maintained at $160^{\circ} \pm 2^{\circ}$ F for 24 hours. After removal of the stack from the oven, the weight shall be removed and the test stack shall be allowed to cool at test room conditions for 30 minutes. The specimens shall be disassembled and examined in sequence for any adhesion or cohesion between adjacent surfaces. If two surfaces appear to be blocked, the free end of one surface shall be vertically clamped so that the other surface hangs down freely. A 200-gram weight shall then be gently attached to the corresponding free end of the second surface. If the two surfaces are not completely separated after two minutes, the specimens are considered blocked. Delamination or rupture of any test surface during separation is also considered blocking.

5.3.1.5 <u>Notes</u>. Procedure letters previously specified in packaging material specifications referencing this test method may be disregarded. The method described herein is intended to replace both Procedure A and Procedure D with one standard test to determine resistance to blocking.

- 5.3.2 <u>Test Method 3005 Contact Corrosivity</u>.
- 5.3.2.1 <u>Scope</u>. This test method details a procedure to determine the corrosive tendencies of packaging materials when in intimate contact with a test surface.
 - 5.3.2.2 Apparatus.
 - 5.3.2.2.1 A circulating air oven that can be maintained at $150^{\circ} \pm 5^{\circ}$ F.
 - 5.3.2.2.2 A desiccator with drying agent.
 - 5.3.2.2.3 An exposure chamber with both temperature and relative humidity controls.
- 5.3.2.2.4 Rectangular steel weights, each measuring 1 by 1 by 3 inches, weighing 0.85 \pm 0.05 pounds.
- 5.3.2.3 <u>Test specimens</u>. Unless otherwise specified, three specimens representative of the material being tested shall be selected at random. When testing flexible sheet material, each test specimen shall measure 2 by 3 inches. When testing cushioning or blocking materials, each specimen size shall be not less than 1 by 3 inches. When testing granular material, each test specimen shall be approximately 20 grams of material ground to a size that passes a U.S. No. 40 sieve, but is retained by a U.S. No. 80 sieve.

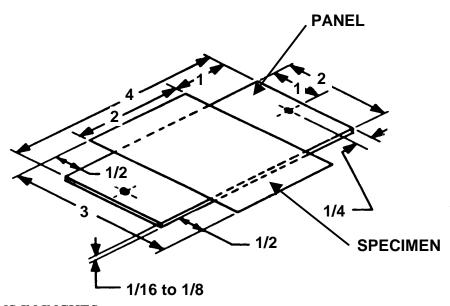
5.3.2.4 <u>Test procedure</u>.

- 5.3.2.4.1 <u>Test panel preparation</u>. The test surface for this evaluation shall be panels, each measuring 1/8 by 2 by 4 inches. Panel material shall be low carbon steel conforming to QQ-S-698, condition 5. Each panel shall be prepared as follows:
 - a. Panels shall be ground to remove surface scale, pits, and other irregularities from all surfaces. One of the large flat surfaces of the panel shall then be finished with 240 grit aluminum oxide or silicon carbide abrasive to a surface roughness of 6 to 12 microinches root mean square (RMS). The polished test surface shall then be wiped with surgical gauze and then scrubbed with a brush or gauze swab in a tank or container of solvent conforming to MIL-PRF-680. This shall be followed by successive immersions in hot mineral spirits, boiling 95 percent methanol, and boiling absolute methanol. Panels shall then be allowed to dry in clean air or placed in a ventilated clean drying oven and then stored in a desiccator

until ready for use. If the panels are stored in the desiccator for a period of more than 24 hours, the latter portion of the surface preparation shall be repeated starting with the 240 grit finishing.

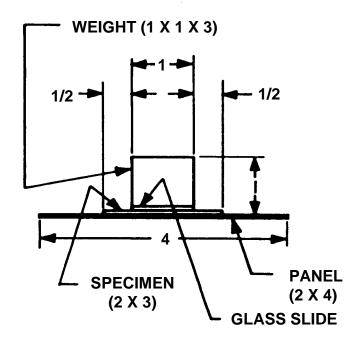
5.3.2.4.2 Procedure.

- 5.3.2.4.2.1 <u>Flexible sheet materials</u>. Each 2 by 3 inch flexible sheet specimen shall be placed across the central portion of the test panel surface, as shown on Figure 2. The edges of the contact area shall be marked on the edges of the panel. A glass slide with a weight superimposed and coinciding shall be centered on top of the specimen with the longitudinal centerline of the slide and weight coinciding with that of the test specimen as shown on Figure 3.
- 5.3.2.4.2.2 <u>Cushioning or blocking materials</u>. Cushioning or blocking material specimens shall be placed across the central portion of the test surface so that the specimen extends over the edges of the panel. Large specimens shall be placed off center so an area of the test surface not less than 2 by 2 inches shall remain uncovered.
- 5.3.2.4.2.3 <u>Granular materials</u>. If granular specimens are being tested, they shall be placed and leveled between parallel lines 1 inch apart across the central portion of the test surface of a panel. The specimen shall be carefully covered with a glass slide, and the steel weight shall then be placed upon the glass slide.
- 5.3.2.4.2.4 Exposure. In all cases the specimen and test surface so arranged shall be exposed for 1/2 hour in air maintained at a temperature of $150^{\circ} \pm 5^{\circ}$ F followed immediately by exposure in air at $120^{\circ} \pm 2^{\circ}$ F and 65 ± 3 percent relative humidity for 20 hours. At the end of the exposure period, the specimens shall be separated from the test surface that shall be immediately examined for evidence of corrosion. Record for each area the one covered by the specimen, and the other not covered whether or not corrosion occurred and a description including the severity and distribution of any corrosion.
- 5.3.2.5 Notes. The given exposure environment and duration are such that corrosion is not visible on unprotected clean steel, but corrosion is visible on the test surface if the material under test has a tendency to induce corrosion. When test surfaces are of other metals, the exposure should be specified. To establish whether or not a specimen induces corrosion of a test surface other than steel, the severity of the test environment and duration of exposure thereto must be sufficient to closely approach incipient corrosive attack of the bare (control) specimen surface. This requires a preliminary test to establish the appropriate duration of exposure.



DIMENSIONS IN INCHES

FIGURE 2. Flexible sheet material specimen placement.



DIMENSIONS IN INCHES

FIGURE 3. Contact corrosivity test assembly.

- 5.3.3 <u>Test Method 3015 Delamination Resistance of Heat-sealable Laminated</u> Materials.
- 5.3.3.1 <u>Scope</u>. This test procedure details a method to determine the delamination resistance of a laminated heat-sealable material in the presence of oil.
 - 5.3.3.2 Apparatus.
 - 5.3.3.2.1 An oven capable of maintaining a temperature of $160^{\circ} \pm 2^{\circ}$ F.
 - 5.3.3.2.2 A heat sealer equipped with controls for temperature, dwell time and pressure.
- 5.3.3.2.3 Two smooth flat metal plates measuring 3 by 6 by 1/4 inches and two 18 pound weights.
 - 5.3.3.2.4 A sufficient quantity of the following test oils to conduct the prescribed tests:
 - a. ASTM Oil No. 3 as specified in ASTM-D471.
 - b. Di-2-ethylhexyl sebacate synthetic oil (available from C. P. Hall Company, Chicago, IL, under trade name of MULTIPLEX DOS).
- 5.3.3.3 <u>Test specimens</u>. Five specimens, each 3 by 6 inches, shall be taken at random from material being evaluated for each test oil.
- 5.3.3.4 Test procedure. Fold each test specimen in half, heat-sealable face to heat-sealable face, to produce a 3 by 3 inch specimen. Make a sharp crease in the specimen by placing the folded specimen between the two smooth flat metal plates and applying one of the 18-pound weights, exerting a pressure of approximately 6 pounds per inch of crease, on top of the fold for 30 seconds. Unfold the specimen and recrease in a similar manner (heat-sealable face to heat-sealable face) at right angles to the first crease by placing the specimen between the two smooth flat plates again and applying both 18-pound weights side by side on top of the fold for 30 seconds. Make a 3 by 3 inch pouch by folding the sample in half along the first crease and heat seal along the two sides. Heat seals shall be 1/2 inch wide. Pour approximately 5 ml of oil into each pouch, carefully keeping the sealing area free from oil and the enclosed air to a minimum, and seal the open end of the pouch. Promptly expose the pouches in an oven maintained at $160^{\circ} \pm 2^{\circ}$ F, for 24 hours by hanging each pouch from the center of its sealed end. Remove pouches from oven. After the pouches return to room conditions (see 4.4), examine the pouches for any oil leakage. Leakage solely through the heat-sealed joints shall be disregarded.

Cut off the three sealed edges and remove all remaining oil. Pull the remaining sheet taut and examine for evidence of swelling, delamination, embrittlement, or other visible defects. A test for delamination of the face film shall be conducted at mid-length of the test specimen by placing the specimen between the thumbs and forefingers so that the thumbs rest on the heat-sealable face. The thumbs shall then be thrust forward and outward in a finger-snapping motion in such a manner that the heat-sealable face will delaminate if it is loosely bonded.

- 5.3.4 <u>Test Method 3027 Water Resistance of Markings.</u>
- 5.3.4.1 <u>Scope</u>. This test procedure details a method to determine the retention of markings on packaging materials after exposure to water.
 - 5.3.4.2 Apparatus.
 - 5.3.4.2.1 Chemically pure water having a pH between 5.5 and 8.0.
- 5.3.4.2.2 A vessel fitted with a means of circulating water at a velocity of approximately one foot per second. The vessel shall have a specimen holding device that will ensure sufficient space between specimens so all markings will be exposed to the circulating water.
- 5.3.4.3 <u>Test specimens</u>. Three specimens shall be tested. Each specimen shall contain one complete set of dry markings selected at random from representative locations on the material being evaluated.
- 5.3.4.4 <u>Test procedure</u>. The test specimens shall be submerged in a vessel filled with water at room temperature for two hours. After removal from the vessel, excess water shall be blotted off the test specimens. They shall then be examined for clarity, legibility, and shall not smear when lightly rubbed with one finger. The examination shall be repeated after the specimens have dried out at room temperature.

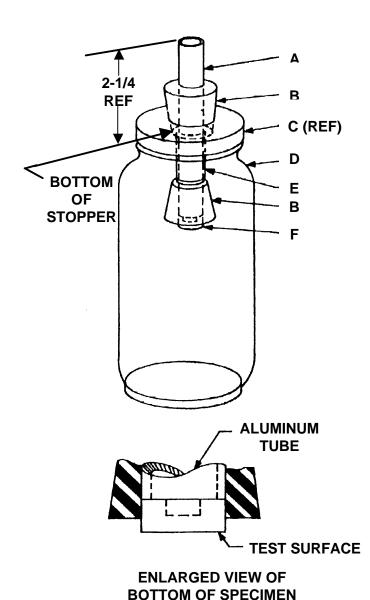
- 5.3.5 <u>Test Method 3028 Water Resistance of Packaging Materials.</u>
- 5.3.5.1 <u>Scope</u>. This test procedure details a method to determine the resistance of packaging material to delamination (ply separation) as a result of exposure to water.
 - 5.3.5.2 Apparatus.
 - 5.3.5.2.1 Tap water.
- 5.3.5.2.2 A vessel capable of accommodating and securing test specimens in a manner to allow unrestricted circulation of water around the specimens.
 - 5.3.5.2.3 An oven capable of maintaining a temperature of $160^{\circ} \pm 2^{\circ}$ F.
- 5.3.5.3 <u>Test specimens</u>. Four specimens, taken at random to adequately represent any variation in the material being evaluated, shall be tested. Specimen size shall be 6 by 8 inches.
- 5.3.5.4 Test procedure. Test specimens shall be immersed in distilled water maintained between 68° and 86° F. The specimens shall be supported in a manner allowing unrestricted circulation of water around the test material. The immersion period shall be 24 hours for fiberboard materials and 48 hours for flexible barrier materials. After removal from the water, specimens shall be blotted dry and examined for ply separation. Flexible barrier materials shall be further exposed in a circulating air oven maintained at $160^{\circ} \pm 2^{\circ}$ F for 24 hours. Re-examine for ply separation after allowing specimens to cool to room temperature.

- 5.3.6 <u>Test Method 3030 Water Vapor Transmission Rate of Barrier Materials.</u>
- 5.3.6.1 <u>Scope</u>. This test procedure details a method to determine the water vapor transmission rate (WVTR) of packaging barrier materials. The test shall be applied to material either as received, after flexing, or after accelerated aging.
 - 5.3.6.2 Apparatus.
- 5.3.6.2.1 A test chamber capable of maintaining a test environment of $100^{\circ} \pm 2^{\circ}$ F and 90 \pm 2 percent relative humidity.
 - 5.3.6.2.2 A heat sealer with controls for temperature, dwell time, and pressure.
 - 5.3.6.2.3 Porous heat-sealable tea bag stock.
- 5.3.6.2.4 Anhydrous calcium chloride desiccant in granular form that passes a No. 8 sieve, but shall be free of fines that pass a No. 30 sieve.
 - 5.3.6.2.5 An analytical balance.
- 5.3.6.3 <u>Test specimens</u>. Unless otherwise specified, four 8 by 12 inch specimens for each test condition shall be selected at random from the sheet material being tested. Two samples shall be taken from each principal direction of the material.
- 5.3.6.4 Test procedure. The test specimens (as received, after flexing, or after aging, as applicable) shall be folded in half and formed into a pouch by making continuous seals along the two 6 inch ends and one 8 inch side using sealing conditions as recommended for the material. Seal widths shall be such that the inside width of the pouch is $5-5/16 \pm 1/16$ inches. A bag to hold the desiccant shall be formed by cutting a 3-1/2-inch wide by 10-1/2-inch long piece from the web of tea bag stock, folding in half to 3-1/2 by 5-1/4 inches, and making a seal not more than 1/4 inch wide along the two sides and the folded edge with the heat sealer. Two such desiccant bags will be required for each test specimen. Fill the desiccant bag with not less than 50 cubic centimeters of the desiccant, and make a closure seal not more than 3/8 inch wide. Insert the desiccant bag into the test specimen pouch and make a closure seal with its inner edge not more than 3/8 inch from the edge so closed. The pouch shall then be exposed in a test chamber maintained at $100^{\circ} \pm 2^{\circ}$ F and 90 ± 2 percent RH for a stabilization period of 16 hours. Remove the pouch from the test chamber and immediately cut off a 1/2 inch wide strip from one end and remove the desiccant bag. Insert a previously weighed fresh desiccant bag into the pouch and close the open end by making a heat seal not more than 3/8 inches wide.

Immediately expose the pouch in the test chamber for a period of 64 to 68 hours. Then remove the test pouch, cut off the closure seal, extract the desiccant bag and immediately reweigh the bag. To avoid the possibility of moisture change during weighings, the desiccant bag should be weighed each time while it is within a closed water vapor-impervious container of known weight. The gain in weight of the desiccant bag during the exposure shall be used to calculate the WVTR for the material being tested.

5.3.6.5 Notes. When testing nonhygroscopic materials which will remain constant in weight during weighing, the following alternate simplified procedure may be used: After the stabilization period, immediately weigh the sealed pouch and promptly begin the exposure period. At the end of the period of exposure, again weigh the sealed pouch and calculate the WVTR for the material being tested.

- 5.4 General physical properties.
- 5.4.1 Test Method 4031 Vapor Inhibiting Ability of VCI Materials.
- 5.4.1.1 <u>Scope</u>. This test method details procedures to determine the corrosion inhibiting effectiveness of volatile corrosion inhibitor (VCI) materials in crystalline or liquid form, or VCI as a coating or treatment on substrate materials.
 - 5.4.1.2 Apparatus.
 - 5.4.1.2.1 A forced draft oven with temperature controls.
 - 5.4.1.2.2 A desiccator with drying agent.
 - 5.4.1.2.3 An atomizer.
 - 5.4.1.2.4 An air source with flow regulator.
 - 5.4.1.2.5 Special apparatus as specified on Figures 4 thru 7.
 - 5.4.1.3 Test specimens.
- 5.4.1.3.1 <u>VCI material in liquid form</u>. For material of this nature, 0.05 ± 0.005 grams of the material being tested shall be weighed and placed in a small vessel. The vessel shall then be placed on the bottom of the test jar.
- 5.4.1.3.2 <u>VCI material in crystalline form</u>. For material of this nature, approximately 1 gram of the material being tested shall be placed in a glass vial which shall be attached to an atomizer. The material shall then be sprayed into the test jar through one of the small holes in the lid until 0.05 ± 0.005 grams, as determined by weight loss, is dispensed.
- 5.4.1.3.3 <u>VCI coated materials</u>. Two strips of the material, each measuring one by six inches, shall be tested for each condition (see 5.4.1.4.3).



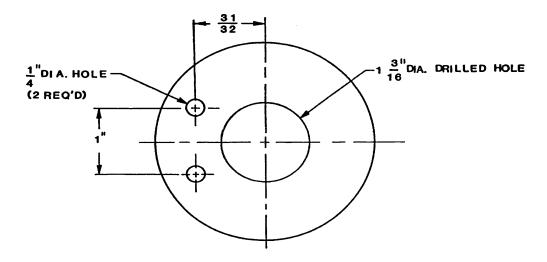
HOLDER ASSEMBLY

- A Aluminum tube 4-1/2 in length, 5/8 OD and 1/2 ID. The tube shall have a capacity of 16 ml of distilled water at 75° ± 3°F.
- B Rubber stopper #6-1/2 rubber stopper with 1/2 hole drilled through center (2 required).
- C Jar lid See Figure 5 for details.
- D Jar Quart size, mouth size 2-3/8 diameter, 7 inches in height, 3-1/4 ID.
- E Insulating sleeve 1/2 ID rubber tubing, length 1-1/2.
- F Test surface 5/8 OD, 1/2 long with 3/8 deep flat bottom hole drilled in center.

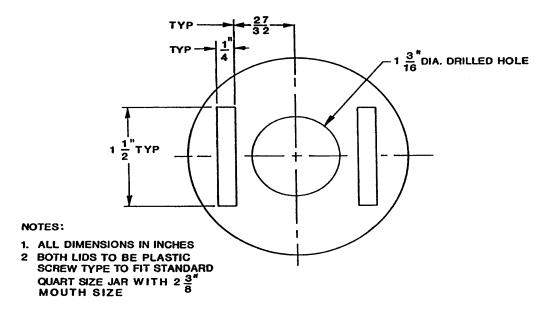
NOTES

- 1. Dimensions in inches.
- 2. All parts of the specimen holder assembly shall be in contact with adjacent part.

FIGURE 4. Test assembly.



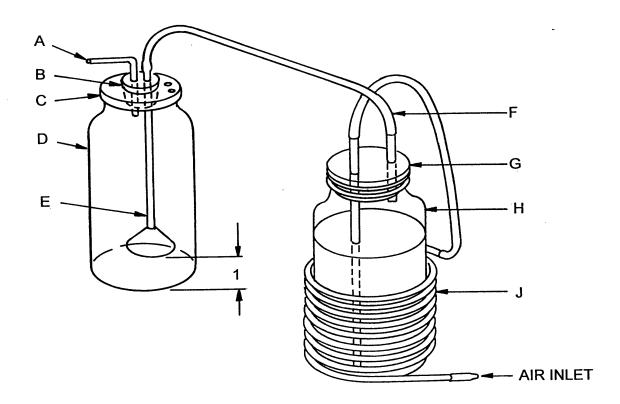
LID FOR TESTING VCI MATERIAL IN CRYSTALLINE OR LIQUID FORM



LID FOR TESTING VCI-COATED MATERIALS

FIGURE 5. Jar lids.

- A Pyrex glass tubing -1/8 ID, length to suit.
- B Rubber stopper No. 6 with two holes to suit insertions.
- C Jar lid See Figure 5.
- D Jar Quart size, 2-3/8 mouth diameter, 7 inches deep, 3-1/4 ID.
- E Pyrex glass funnel Approximately 2 ID at mouth, 8 inches long.
- F Pyrex glass and rubber tubing 1/4 ID and 7/16 OD.
- G Rubber stopper No. 12 with suitable 7/16 dia holes.
- H Quart glass jar -2-3/8 mouth diameter, 7 inches deep, containing glycerine-water solution (Sp. Gr.
 - 1.180) and maintaining the level of solution at 5 inches with the inlet tube immersed 4 inches in solution.
- J Copper coil (OD 1/4, ID 5/32, length 10 ft.) Coil ID diameter 4-3/8.



Notes

- 1. Dimensions in inches.
- 2. After assembly, jar lid shall be sealed to jar with tape.

FIGURE 6. Apparatus for exhaustion of VCI material in crystalline form.

- Pyrex glass and rubber tubing -1/4 ID by 7/16 OD.
- B Rubber stopper No. 12 and No. 13 with suitable 7/16
- C -Hook – stainless steel, 1/16 diameter.
- D Pyrex glass tube -12 by 2-3/8 ID.
- E Diffuser Consisting of glass cylinder (2 OD by 1 high glass tubing) containing glass wool with a surgical gauze covering.
- Quart glass jar -2-3/8 mouth diameter, 7 deep, containing glycerine-water solution (Sp. Gr. 1.180) and maintaining the level of solution at 5 inches with the inlet tube
- Copper coil 1/4 OD by 5/32 ID by 10 feet long, coil ID

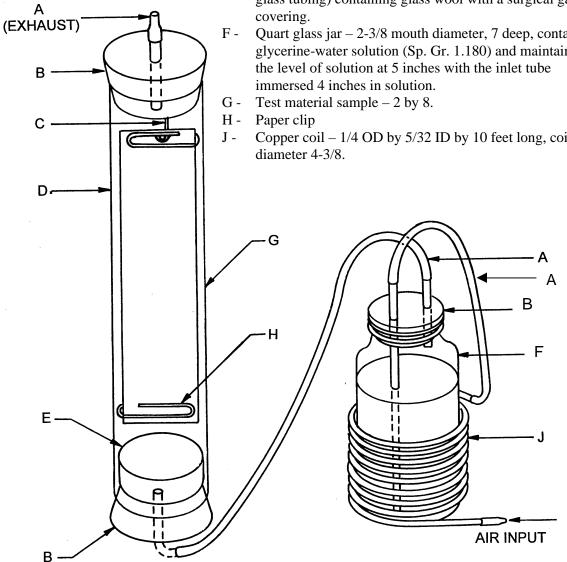


FIGURE 7. Apparatus for exhausting VCI coated material.

5.4.1.4 <u>Test procedure</u>.

5.4.1.4.1 <u>Test surface preparation</u>. The test surface shall be constructed of low carbon steel meeting the requirements of QQ-S-698, Condition 5. A cold finished round bar of this material shall be machined to the dimensions prescribed on Figure 4. The undrilled end shall be surface-ground and polished with 240 grit silicon carbide or aluminum oxide abrasive. The abraded face (test surface) shall then be hand polished with No. 400 aluminum oxide paper at 90° to the previously abraded marks to a surface roughness of 4 to 6 microinches (RMS) for the test surface. (The use of iron oxide abrasives, and any abrasive paper that leaves residues not removable by the following cleaning methods is prohibited. Some abrasive papers and cloths that are intended to be used dry or wet, have been found to leave such residues.) The plug shall be wiped clean with surgical gauze and the test surface examined microscopically for signs of corrosion or other defects. Defective surfaces shall not be used. Each usable metal plug shall be scrubbed with a brush or gauze swab in a tank or container of solvent conforming to MIL-PRF-680. This shall be followed by successive immersions in hot mineral spirits, boiling 95 percent methanol, and boiling absolute methanol, allowed to dry in clean air or placed in a ventilated clean drying oven; and then stored in a desiccator until ready for use. If the plugs are stored in the desiccator for a period of more than 24 hours, the surface preparation shall be repeated starting with the final aluminum oxide polishing.

CAUTION

During and after cleaning, the plugs shall be handled only with clean lint-free gloves, forceps, or other means to prevent contamination of the test surface.

Lens paper shall be used to force the prepared test surface plug into the stopper as shown on Figure 4.

- 5.4.1.4.2 <u>Exhaustion procedures</u>. Some test specimens shall be evaluated in an "aged" state. This condition is referred to as "exhaustion" and test samples for this condition are obtained from VCI material exposed as follows:
- 5.4.1.4.2.1 VCI in crystalline form. The crystalline VCI material shall be introduced into the exhaustion test assembly described on Figure 6 and as detailed in 5.4.1.3.2, except that 0.10 gm ± 0.005 gm of material shall be introduced. The holes in the lid shall be covered with tape. The assembly shall then be connected to the inlet and outlet tubing in order that air maintained at 50 ± 2 percent relative humidity shall pass into the jar containing the VCI at a rate of 100 cubic centimeters per minute. Unless otherwise specified, the completely assembled units shall be placed in a forced draft oven maintained at $100^{\circ} \pm 2^{\circ}$ F for 5 days. The jar containing the VCI

shall be removed from the oven and allowed to cool to room temperature. The procedure to test the exhausted material shall be started immediately.

- 5.4.1.4.2.2 VCI in liquid form. The liquid VCI material shall be introduced into the exhaustion test assembly described on Figure 6 and as detailed in 5.4.1.3.1 except that the large open end of the funnel shall be 2 inches from the bottom of the jar, and 25 cc of material shall be introduced. The exposure shall be conducted as described in 5.4.1.4.2.1 except that the exposure conditions shall be $75^{\circ} \pm 5^{\circ}$ F for 7 days.
- 5.4.1.4.2.3 Non-sealable VCI coated material. Specimens of the material being exhaustion-tested shall be 2 by 8 inches. Treated paper clips shall be placed at each end of the test sample for ease of handling. A 1/8 inch diameter hole shall be punched in the center of each strip approximately 1/8 inch from the top edge. Each test sample shall be placed in the assembly described on Figure 7. An air supply maintained at 50 ± 2 percent RH shall be introduced to the assembly at a rate of 100 cubic centimeters per minute. The entire assembly shall then be placed in a forced draft oven maintained at $140^{\circ} \pm 2^{\circ}$ F for twelve days. The test material shall be removed from the assembly and cut into two 1 by 6 inch specimens for subsequent Vapor Inhibiting Ability testing.
- 5.4.1.4.2.4 <u>Sealable VCI coated material</u>. This exhaustion test is similar to that for non-sealable VCI material except that the test specimen shall be a 1-1/2 by 8 inch pouch created by sealing the edges of a 5 by 8 inch sample of the material under test.
- 5.4.1.4.3 <u>Testing</u>. All tests shall be run in triplicate. In addition, a control sample (blank) shall also be run using a test surface exposed to the same environment with no vapor inhibitor.
- 5.4.1.4.3.1 VCI in crystalline or liquid form (as received). Ten cubic centimeters of a synthetic glycerin-water solution having a specific gravity of 1.076 at $75^{\circ} \pm 3^{\circ}$ F, producing an atmosphere of 90 percent RH shall be introduced into the bottom of the test assembly described on Figure 4. The test specimens, as described in 5.4.1.3.1, shall be added to the test jars. The appropriate lids (see Figure 5) shall be placed on the jars and the holes in the lids and the jar-lid interface shall be sealed with tape. The test assembly shall be exposed to a temperature of $75^{\circ} \pm 5^{\circ}$ F for 20 hours. Sixteen ml of cold water maintained at 40° F shall then be added to the aluminum tubes. After 3 hours, the water shall be removed from the tubes and the test surface shall be examined for evidence of corrosion. If the blank specimen does not show any corrosion, the test shall be rerun.
- 5.4.1.4.3.2 <u>VCI in crystalline or liquid form (after exhaustion)</u>. This test and exposure is similar to the test specified in 5.4.1.4.3.1 except that the jar used for the exhaustion procedure

shall be used as the test assembly by removing the lid with the funnel attachment and replacing it with the appropriate lid specified on Figure 5.

- 5.4.1.4.3.3 <u>VCI in coated material form (as received and after exhaustion)</u>. This test and exposure is similar to the test specified in 5.4.1.4.3.1 except that the two 1 x 6 inch specimens shall be inserted through the slots in the appropriate Figure 5 lid. The specimens shall be held in position by forming a 1/4-inch tab at one end of the specimen. The tab is then taped to the outer surface of the lid. The treated surfaces of the test specimens shall face the test surface.
- 5.4.1.5 <u>Notes</u>. The exposure environment described herein is intended to evaluate the corrosion preventive effectiveness of the VCI material being tested with respect to steel surfaces. For other metals, the exposure conditions and test surface would have to be adjusted to evaluate the effectiveness of VCI material.

- 5.4.2 <u>Test Method 4034 Transparency</u>.
- 5.4.2.1 <u>Scope</u>. This test procedure details a method for determining the degree of transparency of packaging materials.
 - 5.4.2.2 Apparatus.
- 5.4.2.2.1 A legibility standard using 10-point Times Roman black lower case letters and at least ten digits on a white paper background.
- 5.4.2.3 <u>Test specimens</u>. At least three specimens, each 3 by 5 inches, selected at random from the material being tested.
- 5.4.2.4 <u>Test procedure</u>. The characters on the legibility standard shall be examined when viewed through the material being tested when the test material is held three inches away from the legibility standard.

- 5.4.3 <u>Test Method 4046 Electrostatic Properties.</u>
- 5.4.3.1 <u>Scope</u>. This test procedure details a method for determining the electrostatic properties of packaging materials. These properties are determined by measuring the dissipation rate for an electrostatic charge induced on the material being evaluated.
 - 5.4.3.2 Apparatus.
 - 5.4.3.2.1 A 1/8 inch thick aluminum calibration panel measuring 5 x 3 inches.
 - 5.4.3.2.2 A source capable of producing zero to 15 kilovolts, positive and negative.
- 5.4.3.2.3 An electrometer with a full scale reading of 0.01, 0.1, 1.0, 10 and 100, or a recording oscilloscope with a response of 1 microsecond per division, or equivalent.
 - 5.4.3.2.4 An electrostatic test unit, fabricated as illustrated on Figure 8.
- 5.4.3.2.5 A test chamber (see Figure 9) capable of maintaining the test environment specified in 5.4.3.4.2. The chamber shall be provided with hand holes for mounting and removal of test specimens.
- 5.4.3.2.6 A single channel pen type recorder with speeds of 0.5, 1.0, 2.0, 4.0, and 8.0 inches per minute and per second.
 - 5.4.3.2.7 Four RG 114/U cables for use as follows:
 - a. 5 inch cable for the connection between the electrostatic detector and the output connector on the electrostatic test chamber.
 - b. 34 inch cable for connection between the electrostatic test chamber and the electrometer.
 - c. Two 31.5 inch cables for connections between the electrometer and the recorder.
- 5.4.3.2.8 High voltage wire as needed to convey the charge from the high voltage source to the electrostatic test unit and to ground as required.
- 5.4.3.2.9 Three-position control switch for connecting the test specimen to the high voltage source or to ground or to a neutral potential.

- 5.4.3.2.10 The equipment shall be assembled as illustrated on Figure 10.
- 5.4.3.3 <u>Test specimens</u>. Unless otherwise specified, three specimens for each pre-test conditioning shall be selected at random to represent any variation of the material. Each specimen shall be 5 by 3 inches and shall be free of defects such as holes, cracks, and tears. If the specimen is coated, the coating shall be continuous.

5.4.3.4 <u>Test procedure</u>.

- 5.4.3.4.1 <u>Pre-conditioning</u>. Prior to testing, expose three specimens for 12 days in a circulating air oven uniformly maintained at $160^{\circ} \pm 5^{\circ}$ F; three additional specimens in a horizontal position for 24 hours under a continuous water shower; and three more specimens in an atmosphere uniformly maintained at $73^{\circ} \pm 5^{\circ}$ F and 50 ± 5 percent relative humidity.
- 5.4.3.4.2 Test environment. Unless otherwise specified, all specimens shall be placed in the electrostatic test chamber for a minimum of 24 hours immediately before testing. Perform tests in an atmosphere uniformly maintained at $73^{\circ} \pm 5^{\circ}$ F and 12 ± 3 percent relative humidity. This relative humidity can be obtained by inserting sufficient anhydrous calcium chloride into the electrostatic test chamber. The anhydrous calcium chloride shall be replaced as required to maintain required relative humidity.
 - 5.4.3.4.3 Calibration. The test equipment shall be calibrated as follows:
 - a. Turn on all apparatus and allow to warm up, as noted in the operations manual for the particular apparatus.
 - b. On the electrometer, set "multiplier" switch to provide a half-scale reading when the test voltage is applied, the "operate" switch at "zero check," and the meter to read positive charge.
 - c. Adjust the high voltage for 5 KV positive output.
 - d. Mount the 1/8- by 3- by 5-inch aluminum calibration panel between the electrodes in the electrostatic test unit so that the detector head is directly in the center of the panel. Tighten the four wing nuts to secure the panel.
 - e. Set speed of the recorder chart to 1 inch/minute. Turn on recorder.
 - f. Set "operate" switch on the electrometer to "operate."
 - g. Turn three-position control switch to high voltage.

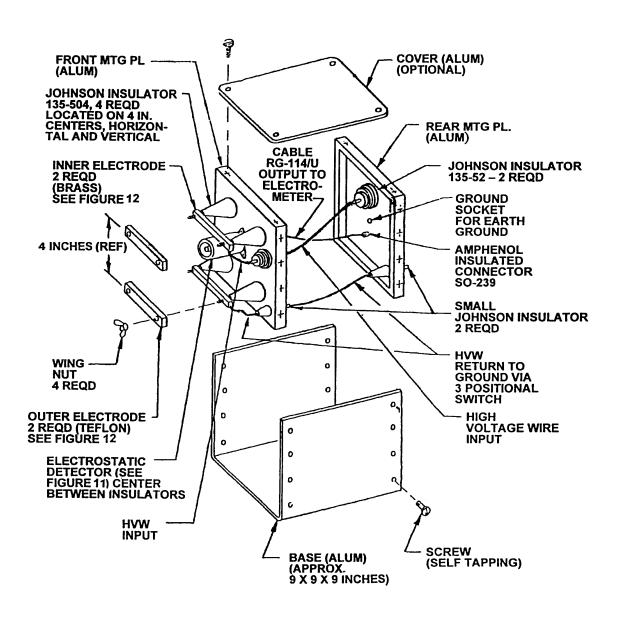
- h. Observe that the reading on the recorder is identical with the measurement on the meter. Adjust the recorder if necessary.
- i. Turn three-position control switch to ground to remove the charge from the test panel.
- j. When the electrometer meter reaches zero, stop the recorder and turn the "operate" switch on the electrometer to "zero check."
- k. Repeat the calibration procedure for 5 KV negative. Set the appropriate controls on the apparatus for negative charge.
- 5.4.3.4.4 <u>Testing</u>. Each conditioned specimen, when tested, shall be mounted vertically between the electrodes and the wing nuts tightened to ensure intimate contact between specimen and electrodes. The test shall then be conducted as follows:
 - a. Set recorder chart speed to 0.5 inch/second and turn on recorder.
 - b. Set electrometer meter switch to indicate positive or negative charge, depending on the high voltage to be applied.
 - c. Adjust the high voltage for the desired 5 KV potential.
 - d. Set "operate" switch on electrometer to "operate."
 - e. Turn the three-position control switch to high voltage.
 - f. When the meter reading stops increasing, indicating the specimen has received its maximum charge, turn the three-position switch to ground position.
 - g. When the meter needle reaches zero or after ten seconds, whichever comes first, stop recorder and move "operate" switch to "zero check."

Each specimen shall be charged three times for both positive and negative charges, allowing specimen to remain grounded for ten minutes after each charging cycle to remove any residual charge on the specimen. For non-homogeneous materials, both surfaces shall be charged by

reversing the face of the material in contact with the inner electrodes. At the conclusion of each charging test, measure the horizontal distance on the recorder chart from the point where the specimen was grounded to the point where the needle reached zero. With the speed of the chart known, the decay time for each specimen in seconds can be calculated.

5.4.3.5 <u>Notes</u>.

- 5.4.3.5.1 The purpose of this procedure is to evaluate the electrostatic buildup and dissipation properties of packaging materials used to protect electronic parts that are susceptible to damage by electrostatic discharge.
- 5.4.3.5.2 A Keithley 621 Electrometer may be used. Other settings may apply if another suitable electrometer of different design is used.



NOTE: AFTER ASSEMBLY, THE BRASS DISC OF THE ELECTROSTATIC DETECTOR SHALL BE ADJUSTED SO THAT THE DISTANCE BETWEEN THE DISC AND A MOUNTED SPECIMEN IS APPROXIMATELY ONE INCH.

FIGURE 8. Electrostatic test unit.

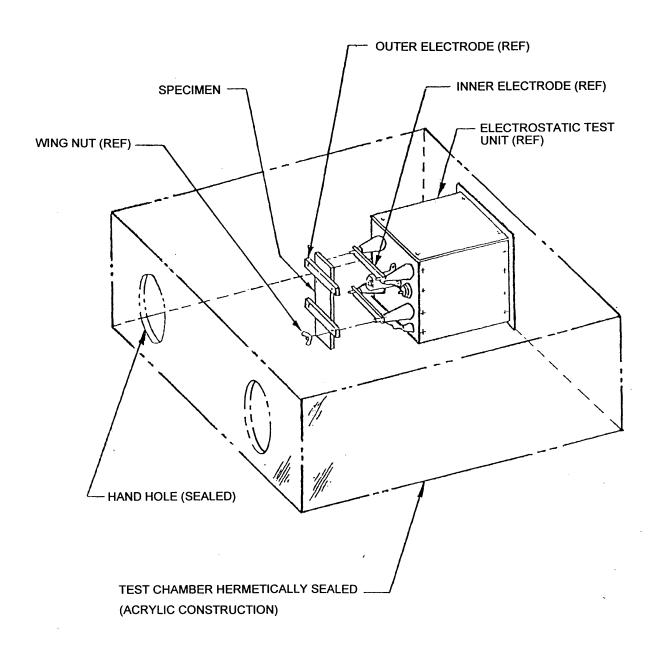


FIGURE 9. Electrostatic test chamber.

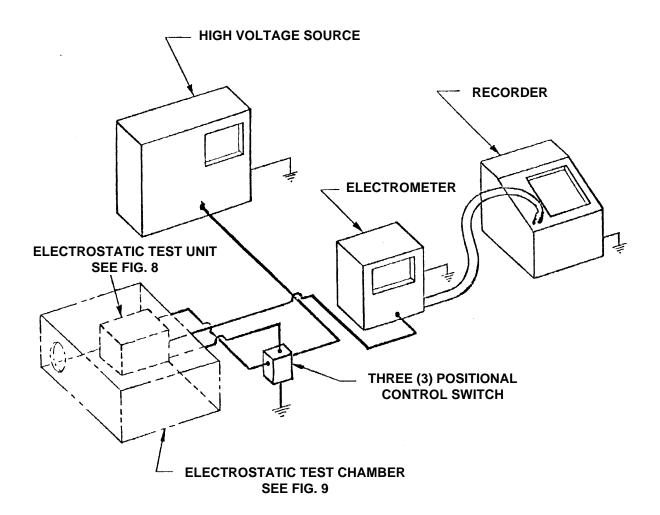


FIGURE 10. Electrostatic test arrangement.

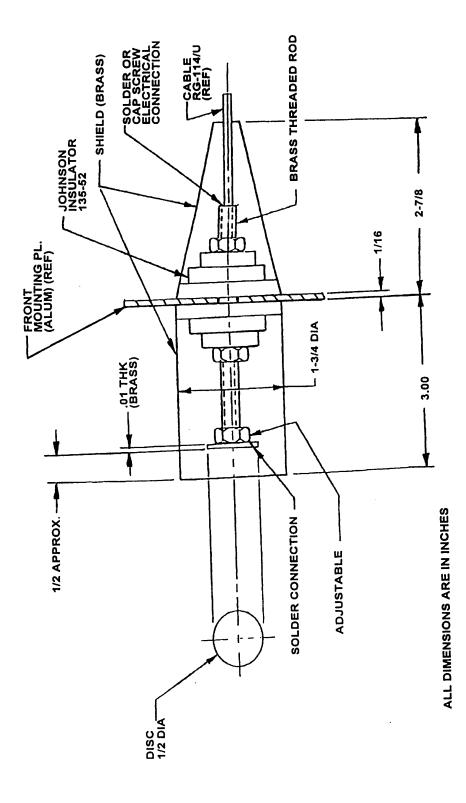
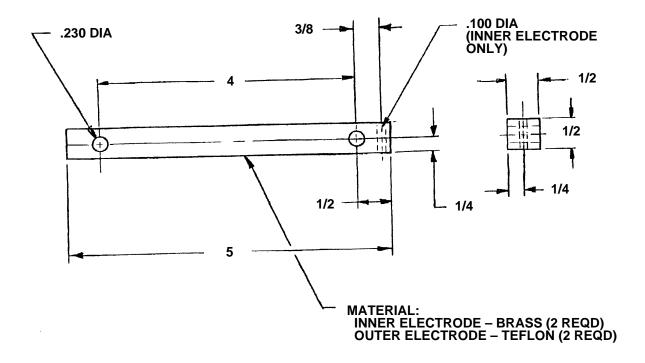


FIGURE 11. Electrostatic detector.



ALL DIMENSIONS ARE IN INCHES

FIGURE 12. Electrode.

- 5.5 <u>Properties of containers</u>.
- 5.5.1 Test Method 5009 Leaks in Containers.
- 5.5.1.1 <u>Scope</u>. This test procedure details methods to determine leakage of air or liquids from sealed containers. Five test methods are defined; choice of method is determined by the physical characteristics of the container being evaluated (see 5.5.1.5).
 - 5.5.1.2 Apparatus.
- 5.5.1.2.1 Vessels of sufficient size to allow complete submergence of the container being tested.
- 5.5.1.2.2 A vacuum pump and pressure gage if the vacuum retention test is being conducted.
- 5.5.1.2.3 A pressure gage and a supply of compressed air if the pneumatic pressure test is being conducted.
- 5.5.1.3 <u>Test specimens</u>. Unless otherwise specified, a specimen shall be one container and contents (actual or simulated), packed and sealed as for shipment.
 - 5.5.1.4 Test procedures.
 - 5.5.1.4.1 Vacuum retention technique.
- 5.5.1.4.1.1 Specimen preparation. Provisions shall be made for connection of a tube to evacuate air and installation of a gage to indicate any loss in vacuum pressure. Such provisions shall be a tube and gage sealed into openings at the corners of a seam of a flexible container or a drilled and tapped hole for a plug or a valve stem incorporated in a rigid container or other acceptable devices that can be either sealed or removed from the container without adverse effect.
- 5.5.1.4.1.2 Testing. Connect the vacuum pump to the specimen and evacuate the air until the desired vacuum is attained. Unless otherwise specified, the vacuum pressure shall be 9 \pm 1 mm of mercury or $5\pm1/2$ inches of water. The required vacuum may be drawn more than once to ensure that equilibrium within the specimen is reached. When the specimen is evacuated to a constant specified pressure, stop evacuating air and record the vacuum pressure gage reading. After ten minutes, the gage shall be read again to determine if there is a loss in vacuum pressure, indicating a leak.

- 5.5.1.4.2 <u>Pneumatic pressure technique</u>.
- 5.5.1.4.2.1 Specimen preparation. Provisions shall be made for connecting a tube or clamp-in valve to the specimen. Either attach a pressure gage to the specimen or use a low-pressure hand type tire gage to sense any loss in pressure. A tube or valve shall be sealed into an opening at one end of a seam in a flexible container, a hole drilled and tapped with a plug or a "clamp-in valve" stem incorporated in a rigid container, or other methods that will permit removal and seal without adverse effects of the serviceability of the container.
- 5.5.1.4.2.2 <u>Testing</u>. Pressurize the specimen with air from compressed air supply. Gradually introduce air until either the prescribed pressure in the specimen is attained or leakage becomes apparent. Unless otherwise specified, the pressure (P) shall be calculated equal to the specified strength of the barrier seams (S) times pi (π) divided by the sum of the two smaller dimensions (d1 and d2) of the package.

$$P = \frac{S \times \mathbf{p}}{(d1 + d2)}$$

(For example, to test a package 10 by 6 by 4 inches enclosed in MIL-PRF-131 barrier material, the pressure shall be the specified strength of MIL-PRF-131 barrier seam (3-1/2 lb./in.) times pi (3.14) divided by the sum of the smaller dimensions (6 + 4 inches); that is, the pressure shall be 11/10 or 1.1 lb./sq. in. For other sizes other pressures shall be calculated in a similar manner or read from an appropriate curve.)

CAUTION

Pneumatic pressure may cause explosive failure of weak specimens. The applied pressure should be no greater than necessary to reveal leaks.

When the specimen is pressurized to a constant specified pressure, read and record this initial pressure. After 30 minutes, read and record the final gage pressure. If no change is noted between the initial and final gage pressure, the item is considered satisfactorily sealed.

- 5.5.1.4.3 Squeeze technique. (Applicable only to flexible specimens)
- 5.5.1.4.3.1 <u>Specimen preparation</u>. During final sealing of the specimen, entrap as much air as possible within the specimen.

- 5.5.1.4.3.2 <u>Testing</u>. Either submerge the specimen 1 to 2 inches under water and, while squeezing the specimen to force air to the area under observation, observe all seams and surface for leakage; or coat all seams, joints, or other areas likely to leak with a bubble-supporting film and observe each for leaks while squeezing the specimen to force air to the area under observation.
- 5.5.1.4.4 <u>Hot water technique</u>. Unless otherwise specified, submerge the specimen in water heated to a temperature at least 50°F above the initial temperature of the specimen (not over 110°F for wax-dipped specimens). While holding the specimen submerged with the uppermost surface covered by not more than 1 inch of water, observe for at least 15 seconds to detect leakage. The specimen shall be rotated and observed repeatedly until all of the specimen has been examined. Total time in hot water shall not exceed 8 minutes.
- 5.5.1.4.5 <u>Submersion (or immersion) techniques</u>. Unless otherwise specified, the specimen shall be submerged so that the uppermost surface is beneath the water surface not less than 1 inch or more than 2 inches for 1 hour or longer in water maintained at a temperature at least 40°F below the temperature at which the specimen is sealed. After submersion and before opening the specimen, carefully dry the outside of the specimen where the opening will be made. Then open the specimen and inspect the inside for leakage.

5.5.1.5 Notes.

5.5.1.5.1 Selection of technique. The most appropriate technique will depend principally upon the construction, size and weight of the unit pack and the information needed. The hot water technique is appropriate for large unit packs. The squeeze technique is appropriate for small unit packs constructed of flexible materials such as plastic film. The vacuum retention technique does not specifically locate leaks and may not indicate the existence of tiny leaks in a large unit pack. The submersion (or immersion) technique for detecting water leakage is not as sensitive as the air leakage tests, but it is appropriate to reveal whether or not water might leak into the unit packs and, depending upon the duration of the test, gives some indication of the extent to which the materials used in the pack are waterproof. The pneumatic pressure technique is primarily appropriate for rigid containers. Neither the hot water nor the pneumatic pressure techniques are appropriate for rigid containers that are sealed with tapes; the submersion technique shall be used.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

- 6.1 <u>Intended use</u>. This standard is a listing of uniform test methods that have been developed to evaluate properties of materials used in military packaging applications. It is intended that the test methods detailed in this standard be referenced (by number) in packaging material specifications. This eliminates the need to repetitively detail any standard test method in each material specification.
- 6.2 <u>Issue of DoDISS</u>. When this standard is used in acquisition, the applicable issue of the DoDISS should be cited in the solicitation (see 2.2 and 2.3).

6.3 <u>Subject term (keyword) listing.</u>

Blocking resistance Leakage

Contact corrosivity Volatile corrosion inhibitors

Curl resistance Seam strength
Delamination Test methods

Electrostatic properties Water vapor transmission rate

Flex testing

6.4 <u>Changes from previous document</u>. Marginal notations are not used in this standard to identify changes with respect to FED-STD-101C due to the extensiveness of the changes.

APPENDIX A

CHRONOLOGY OF PACKAGING MATERIALS TEST METHODS

- A.1 <u>SCOPE</u>. This appendix provides a cross reference and tracks test methods of government packaging test methods. This Appendix is not a mandatory part of this standard. The information contained herein is intended for guidance only.
- A.2 <u>APPLICABLE DOCUMENTS</u>. This section is not applicable to this appendix.
- A.3 <u>HISTORY</u>. This Military Standard supersedes FED-STD-101C. The latter standard originally contained approximately 250 test methods for evaluation of military packaging materials. This number has been steadily reduced as a result of supersession by non-Government documents and deletion of methods as a result of little or no usage thereof.

A.4 <u>CHRONOLOGICAL SUMMARY</u>. To assist users following up references to test methods specified in FED-STD-101, all test methods that were listed in FED-STD-101C and FED-STD-101B are listed in Table A.I. Disposition of each of these methods is identified as well as a cross reference to the 3-digit test method identifier that was used in FED-STD-101A. All test methods listed in Table A.I that are dispositioned as "deleted" have been removed from the system over the years with no superseding information. Any document specifying these deleted test methods should be updated to reflect currently available alternative military or industry test method standards.

TABLE A.I. <u>Test methods chronological summary</u>.

FED-STD-101B and/or				Cross-reference to FED-STD-101A
FED-STD-101C	Dist	oosition	Superseding	3-Digit Test Method
Test Method	Deleted	Superseded	<u>Document</u>	_
1001	X			238
1002	X			316
1003		X	MIL-STD-3010	332
1004		X	ASTM-D3652	
2001	X			160
2002	X			255
2003	X			244
2004	X			162
2005	X			230
2006	X			231
2007		X	*TAPPI T810 or	250
			*TAPPI T807	
2008		X	ASTM-F36	292
2009	X			293
2010	X			294
2011	X			296
2012		X	ASTM-D695	297
2013		X	ASTM-D2221	300
2014	X			165
2015		X	MIL-STD-3010	183
2016	X			304
2017		X	MIL-STD-3010	308
2018		X	ASTM-D1098	309
2019		X	ASTM-D790	310
2020		X	ASTM-D3499	311
			ASTM-D3500	
			ASTM-D3501	
			ASTM-D3502	
			ASTM-D3503	

^{* =} Document is not DOD adopted.

TABLE A.I. <u>Test methods chronological summary</u> – Continued.

FED-STD-101B				Cross-reference						
and/or	ъ.	•.•	G 11	to FED-STD-101A						
FED-STD-101C		position	Superseding	3-Digit Test Method						
Test Method	<u>Deleted</u>	<u>Superseded</u>	<u>Document</u>							
2021		**	. CEN. 1 D 1005	212						
2021		X	ASTM-D1037	312						
2022	X	X	ASTM-D1238	170						
2023	X			242						
2024		X	MIL-STD-3010	259						
2025	X			313						
2027	X			110						
2028	X			340						
2029	X			155						
2030		X	ASTM-D3499	265						
			ASTM-D3500							
			ASTM-D3501							
			ASTM-D3502							
			ASTM-D3503							
2031	X			112						
2032	\mathbf{X}			321						
2033		X	ASTM-D2808	348						
2034	X			234						
2035	X			326						
2036		X	*TAPPI T414	327						
2037	X			248						
2038	X			246						
2039	X			247						
2040		X	ASTM-D638	245						
2041	X			225						
2042	X			228						
2043	X			224						
2044	11	X	ASTM-D3953	227						
2045		4 1	1101111 100700							
2010	X			226						

^{* =} Document is not DOD adopted.

TABLE A.I. <u>Test methods chronological summary</u> – Continued.

FED-STD-101B and/or				Cross-reference to FED-STD-101A
FED-STD-101C	Dier	osition	Superseding	3-Digit Test Method
Test Method	Deleted	Superseded	Document	<u>5 Digit Test Method</u>
10001111000	Defeted	Buperseded	Document	
2047	X			218
2048	X			107
2049	X			158
2050		X	ASTM-D3330	
2051		X	ASTM-D3889	
2052	X			
2053		X	ASTM-D3813	
2054		X	ASTM-D3654	
2055		X	ASTM-D3654	
2056	X			
2057	X			
2058		X	ASTM-D3662	
2059	X			
2060	X			
2061		X	ASTM-D3759	
2062		X	ASTM-D3759	
2063		X	ASTM-D3759	
2064		X	ASTM-D3811	
2065		X	MIL-STD-3010	
2071	X			
3001	X			254
3002	X			337
3003		X	MIL-STD-3010	223
3004	X			341
3005		X	MIL-STD-3010	338
3006	X			339
3007	X			299
3008	X			229
3009	X			192

TABLE A.I. <u>Test methods chronological summary</u> – Continued.

FED-STD-101B and/or				Cross-reference to FED-STD-101A					
FED-STD-101C	Dic	position	Superseding	3-Digit Test Method					
Test Method	Deleted	Superseded	Document	5-Digit Test Method					
<u>rest wiethod</u>	Deleteu	Superseded	Document						
3010	X			305					
3011	X			163					
3013	X			167					
3014	X			306					
3015		X	MIL-STD-3010	260					
3016	X			261					
3017	X			262					
3018	X			187					
3019	X			266					
3020	X			233					
3021	X			125					
3022		X	ASTM-D779	270					
3023	X			269					
3024	X			268					
3025	X			274					
3026	X			329					
3027		X	MIL-STD-3010	285					
3028		X	MIL-STD-3010	345					
3029		X	ASTM-D724	106					
3030		X	MIL-STD-3010	286					
3031	X			333					
3032		X	ASTM-D3816						
3033		X	ASTM-D3833						
4001		X	ASTM-D4157	287					
			ASTM-D4158						
4002	X			219					
4003	X			289					
4004	X		MIL-STD-2073-1	290					
4005		X	ASTM-D1894	291					
4006	X			256					
4007	X			298					

TABLE A.I. <u>Test methods chronological summary</u> – Continued.

FED-STD-101B and/or				Cross-reference to FED-STD-101A							
FED-STD-101C	<u>Dis</u>	<u>position</u>	Superseding	3-Digit Test Method							
Test Method	<u>Deleted</u>	<u>Superseded</u>	<u>Document</u>								
4008	X			301							
4009		X	ASTM-D1531	303							
4010	X			222							
4011	X			257							
4012		X	ASTM-D1310	307							
4013	X			177							
4014	X			235							
4015	X			131							
4016	X			315							
4017	X			317							
4018		X	ASTM-E462	220							
			ASTM-E619								
4019	X			174							
4020	X			263							
4023		X	ASTM-C148	320							
4024	X			173							
4025	X			267							
4026		X	ASTM-B117	322							
4027	X			323							
4029	X			123							
4030	X			134							
4031		X	MIL-STD-3010	344							
4032		X	ASTM-D1545	280							
			ASTM-D562								
4033	X			281							
4034		X	MIL-STD-3010	282							
4035	X			283							
4036	- -	X	ASTM-D570	284							
4037	X			271							
4038	X			272							

 $TABLE\ A.I.\ \ \underline{Test\ methods\ chronological\ summary}-Continued.$

FED-STD-101B and/or FED-STD-101C		position	Superseding	Cross-reference to FED-STD-101A 3-Digit Test Method					
Test Method	<u>Deleted</u>	<u>Superseded</u>	<u>Document</u>						
4020	W			225					
4039	X			325					
4040	X			133					
4041	X			273					
4042	X	T 7	. CT)	275					
4043		X	ASTM-C177	185					
4044	X			276					
4045		X	ASTM-D1003	277					
4046		X	MIL-STD-3010	347					
4047		X	ASTM-D3611						
4048	X								
4049	X								
4050		X	ASTM-D3815						
5001	X			217					
5002	X			335					
5003		X	ASTM-D642	295					
5004	X			168					
5005		X	ASTM-D4169	214					
5006	X			302					
5007		X	ASTM-D4169	216					
5008		X	ASTM-D4169	213					
5009		X	MIL-STD-3010	241					
5010	X			314					
5011	11	X	ASTM-D4169	236					
5012		X	ASTM-D4169	212					
5013	X	11	7101111 107	243					
5014	71	X	ASTM-D4169	240					
5015	X	21	TANTINI D'TIU)	239					
5016	11	X	ASTM-D4169	209					
5017		X	ASTM-D4169	210					
5017		X	ASTM-D4169 ASTM-D4169	215					
		X							
5019		Λ	ASTM-D4169	278					

TABLE A.I. <u>Test methods chronological summary</u> – Continued.

FED-STD-101B				Cross-reference					
and/or				to FED-STD-101A					
FED-STD-101C	<u>Disp</u>	<u>oosition</u>	Superseding	3-Digit Test Method					
Test Method	<u>Deleted</u>	<u>Superseded</u>	<u>Document</u>						
5020		X	ASTM-D4169	279					
5021		X	ASTM-D1008	252					
			ASTM-D895						
5022		X	*TAPPI T410	204					
5023		X	ASTM-D4169	211					
5024		X	*TAPPI T400	205					
5025	X			319					
5026	X			264					
5027	X			208					
6001	X			253					
6002	X			203					
6003	X			129					
6004	X			135					
6005	X			206					
6006	X			207					
6007	X			126					
6009	X			288					
6010	X			144					
6011	X			237					
6012	X			157					
6013		X	ASTM-D1030	249					
6014	X			318					
6017	X			221					
6018	X			232					
6019	X			251					
6020		X	ASTM-D549	324					
6021	X			330					
6022	X			331					
6023	X			349					

^{* =} Document is not DOD adopted.

CONCLUDING MATERIAL

Custodians:

Army-SM

Navy – AS

Air Force – 99

Preparing activity:

Navy – AS

(Project PACK-1131)

Review activities:

Army – AR, AV, CR3, GL3, MI, MR

Navy – OS, SA, SH, YD

Air Force – 11

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INSTRUCTIONS

- 1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
- 2. The submitter of this form must complete blocks 4, 5, 6, and 7, and send to preparing activity.
- 3. The preparing activity must provide a reply within 30 days from receipt of the form.

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3. DOCUMENT TITLE TEST PROCEDURES I	FOR PACKAGING MATER	RIALS								
4. NATURE OF CHANGE (Identify paragr	aph number and include proposed rev	vrite, if possible. Attach extra sh	eets as needed.)							
5. REASON FOR RECOMMENDATION										
6. SUBMITTER a. NAME (Last, First, Middle Initial)	1	b. ORGANIZATION								
c. ADDRESS (Include ZIP Code)		d. TELEPHONE (Include Area Code) (1) Commercial (2) DSN (If applicable) 7. DATE SUBMITTI (YYYYMMDD)								
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